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Sliding wear of YTZP ceramic against steel: observations on ceramic transfer and wear transition

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Sliding tests were conducted, in air, of YTZP ceramic pins against steel discs at an applied pressure of 15.5 MPa over a speed range of 0.3 to 4.0 $\mathrm{m\,s^{-1}}$. Pin wear was not detectable until 2.0 $\mathrm{m\,s^{-1}}$, after which a finite but small wear rate was observed at 3.0 $\mathrm{m\,s^{-1}}$, accompanied by a red glow at the contacting surface. A transition in wear behaviour and friction (μ) occurred at 4.0 $\mathrm{m\,s^{-1}}$, increasing the former by over two orders of magnitude. Both μ and wear behaviour changed with time at 4.0 $\mathrm{m\,s^{-1}}$. During initial periods μ was high and wear rate increased steadily with time accompanied by ceramic transfer onto the disc, which increased with time. When disc coverage exceeds a certain threshold value, μ decreased rapidly and the wear rate stabilized at a very high value. Metal transfer was not observed at any speed. High surface temperatures brought about significant adhesion between TZP and steel and this together with enhanced plastic deformation brought about a transition in wear behaviour.

1. Introduction

Ceramics are better candidates for many tribological applications than metals. Their major drawback is inherent lack of toughness and very limited plastic deformation. These have been overcome to some extent by the discovery of stabilization of zirconia by refractory oxides which has resulted in enhancement of toughness due to transformation toughening [1, 2] and microcrack toughening [3]. The contribution of stress-induced transformation is expected to decrease with temperature and disappear altogether above 900 °C, at which temperature embedded particles of monoclinic zirconia transform into a stable tetragonal form and there is no driving force for the tetragonal–monoclinic (t → m) transformation. But high strength and toughness have been observed at temperatures higher than 900 °C [4]. One explanation for this high toughness at high temperatures is expressed in terms of ferroelastic domain switching [5].

Considering the superior toughness exhibited, particularly in the case of TZP, many authors have tried to correlate the wear resistance to toughness rather than hardness. However, there does not appear to be a straightforward relationship as different authors reported contradictory evidences. On one hand Fischer *et al.* [6] reported that during self-mated sliding of yttria stabilized zirconia, with different fracture toughnesses, the wear resistance is proportional to the fourth power of toughness. On the other hand, according to Zum Gahr *et al.* [7] the fracture toughness increases with increase in grain size, and increase in grain size increases the incidence of intergranular fracture which in turn increases the wear. Further, Bundschuh and Zum Gahr [8] have reported an increase in wear with porosity even though fracture toughness remains the same. The t → m phase transformation has been reported to occur during abrasive wear [9, 10] but not in self-mated sliding [11] or metal cutting [12].

Similar to other structural ceramics, where sliding against metals is often reported to be controlled by large metal transfer, metal transfer has been reported at room temperature [13] as well as high temperatures in the case of TZP sliding against metals [14]. Both Stachowiak and Stachowiak [14] and Gates *et al.* [15] observed an increase in friction with increase in temperature due to enhanced metal transfer. Demizu *et al.* [16], for the sliding of YTZP against metals with varying surface chemical activity, have reported that metals with higher chemical activity (Gibbs free energy of ionization) exhibit metal transfer giving rise to higher friction, whereas those with low chemical activity show less metal transfer and low friction. In the case of YTZP [17] and Ce–TZP [15] very little iron transfer has been reported and in the latter case the possibility of a tribochemical interaction has been suggested.

The present paper reports the findings on the wear of YTZP sliding against steel, carried out in a pin on disc machine, as a function of speed at a constant pressure of 15.5 MPa. The questions that arise based on the literature are whether any wear of YTZP occurs at all particularly in view of the metal transfer reported. Further, the nature of the tribochemical interaction products formed and role of phase transformation during wear is explored. In this context the wear process of YTZP against steel as a function of speed has been discussed.

2. Experimental procedure and material used

Rods of (6 mm diameter) yttria doped (3 mol.%) zirconia (YTZP) (obtained from M/s CVC Scientific Products Ltd., Berkshire, U.K) was used in the present work. One end of these rods was cut into a step of contact area 3.0 mm^2 (1.5 mm × 2.0 mm) using a slow speed diamond saw. Experiments were conducted on a pin-on-disc machine, by sliding the YTZP ceramic pin against a En-24 (AISI 4340) steel disc (by keeping 2.0 mm side parallel to the sliding direction) at a pressure of 15.5 MPa over a speed range of 0.3 to 4.0 m s^{-1} (at 0.3, 0.5, 0.75, 1.0, 1.5, 2.0, 3.0 and 4.0 m s^{-1}) under ambient conditions of 40–70% RH and at room temperature (25 °C). The composition of the steel disc is given in Table I. It had a hardness of 50 HRc and surface roughness of 0.3 μm CLA. The highest speed employed was an experimental limitation, as will be discussed later.

Each experiment was preceded by 5 min of running-in against a fresh 600 grit emery paper (fixed on the disc) at a pressure of 3.3 MPa and a speed of 0.1 m s^{-1}. After running-in, without disturbing the pin from the holder, both pin and disc were thoroughly cleaned with acetone. Sliding was started at a pressure of 15.5 MPa and a speed of 0.3 m s^{-1} and was continued until both friction and wear recordings showed a steady state. Once the steady state was reached, sliding was stopped, debris were collected (stored in a vacuum desiccator for further analysis) and the disc was allowed to cool to room temperature. The sliding was then continued at the next higher speed of 0.5 m s^{-1} using the same pin and disc track until steady state was reached. In the same manner the sliding was continued until a speed of 4.0 m s^{-1} was reached. Four such sets of experiments, each starting with a run-in pin and a fresh disc track, were carried out. Since these experiments involved change of speed for the same pin and disc track, independent experiments using a fresh pin and disc track were carried out at 0.5, 1.5, 2.0, 3.0 and 4.0 m s^{-1}. In these experiments, along with the wear measurements obtained from LVDT, the profiles of disc tracks were also obtained to get an estimation of disc wear.

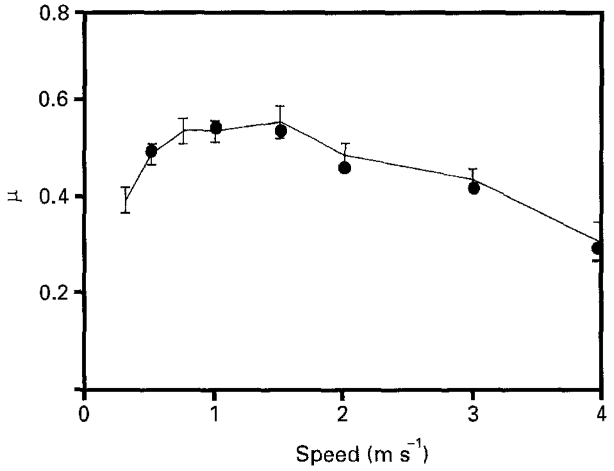

Figure 1 Variation in coefficient of friction (μ) with speed. Filled circles correspond to data from the direct experiments.

TABLE I Chemical composition of AISI 4340 steel disc by weight percentage

C	0.39
Mn	0.62
Si	0.22
S	0.023
P	0.023
Ni	1.4
Cr	1.02
Mo	0.02
Fe	balance

3. Results

Variation in average coefficient of friction and wear rate of the pin with speed are given in Figs 1 and 2 respectively. Results of the fresh pin and disc experiments are shown by filled circles. It can be seen that these points are very close to those obtained from the continuous sliding experiments. Friction initially increased slightly to achieve a value of about 0.5; beyond 2.0 m s^{-1} there was a gradual decrease. For sliding at low speeds the pin wear was not measurable. Measurable wear occurred only at 3.0 m s^{-1}. When the speed was increased to 4.0 m s^{-1}, the wear rate increased by more than two orders of magnitude. At this speed, the wear rate increased with sliding distance as shown in Fig. 3. The sudden increase in wear is associated with transfer of pin material onto the disc and any further sliding at 4.0 m s^{-1} or higher speeds was not possible because of very high wear rate. In Fig. 2 the wear rate corresponding to 4.0 m s^{-1} is the average obtained over a sliding distance of 3500 m. At the same speed the changes in μ showed an opposite trend. Initially it was high and subsequently changed to a low value

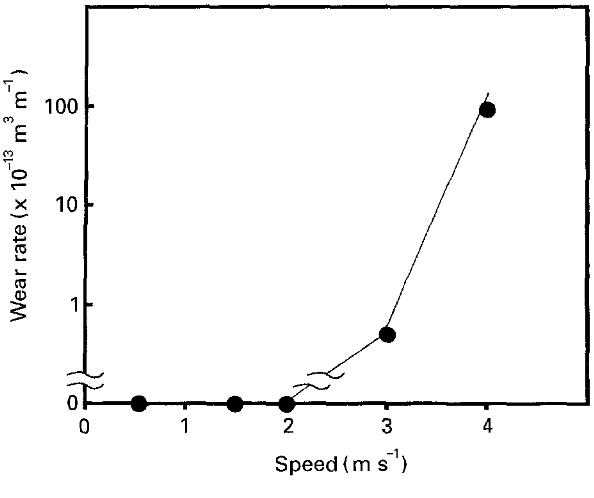

Figure 2 Variation in wear rate with speed. Filled circles correspond to the results from the direct experiments.

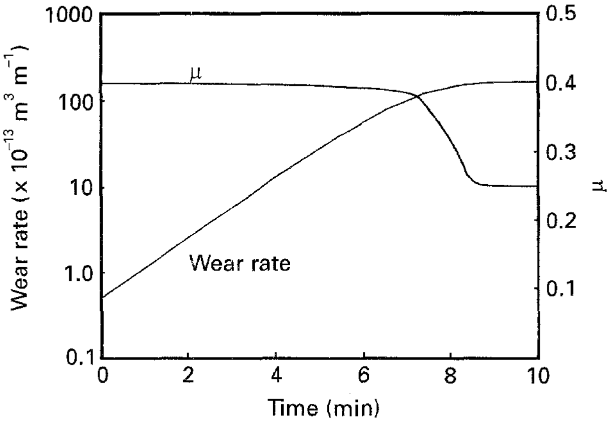

Figure 3 Variation in coefficient of friction (μ) and wear rate with time at 4.0 m s^{-1}.

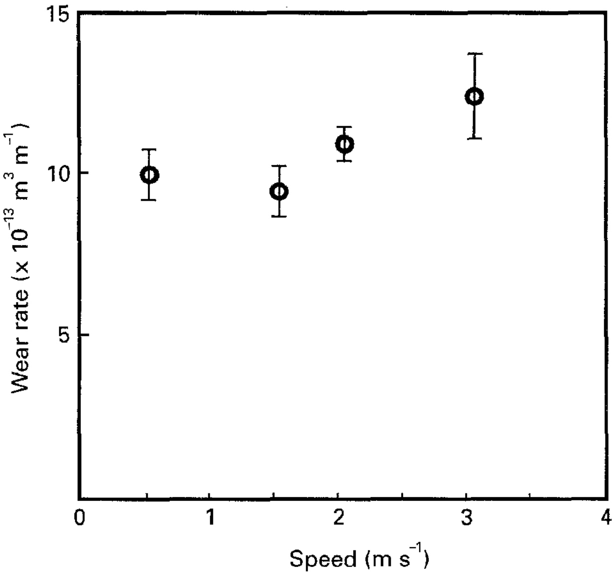

Figure 4 Variation in disc wear rate with speed.

when the wear was very high and remained in that state during the rest of the sliding (Fig. 3).

Fig. 4 shows disc wear at selected speeds. The wear rate of the disc, as calculated by profilometric measurements showed a small initial decrease between 0.5 and 1.5 m s^{-1} followed by an increase between 1.5 and 3.0 m s^{-1}.

3.1. Visual observations

In wear tests performed up to a speed of 1.5 m s^{-1} the disc surface appeared brown and at 2.0 m s^{-1} it turned to a bright metallic steel colour. At 3.0 m s^{-1} black patches were observed on the disc track. At 4.0 m s^{-1} a few thick lumpy deposits, presumed to have come from the pin could be observed on the disc surface along with the black patches. At the same speed when the wear rate further increased, the disc was covered predominantly by the deposits and the black patches were not present.

Correspondingly the pin surface at low speeds appeared faintly brown but became increasingly whitish with increase in speed until 1.5 m s^{-1}. At 2.0 m s^{-1} the pin surface also appeared slightly blackish and at 3.0 m s^{-1} it became completely black and appeared to be covered with a uniform thin layer. At 4.0 m s^{-1} the pin surface became white and remained so throughout the remainder of the sliding.

Above a sliding speed of 3.0 m s^{-1} a red glow could be observed at the contact interface between the pin and the disc. An intense yellowish glow was observed during sliding and it extended into the pin up to about 5 mm from the interface at a sliding speed of 4.0 m s^{-1}.

3.2. Scanning electron microscopy (SEM) studies

Figs. 5 (a–j) present SEM micrographs of the run-in and worn pin surfaces at various selected speeds. The run-in surface (Fig. 5(a)) shows a number of pits on a finely grooved surface. The low magnification view of the surface worn at 0.5 m s^{-1} (Fig. 5(b)) appears very similar to the run-in surface suggesting the occurrence of very small, if any, wear. The high magnification view of the same surface in Fig. 5(c) shows a granular appearance of pits suggesting grain pullout. The smooth regions, show a certain degree of grain relief. At 1.5 m s^{-1} the pin surface appears completely smooth (Fig. 5(d)) suggesting that with increase in speed, the surface pits were progressively removed from the pin surface. A high magnification view of the same surface shows grain relief (Fig. 5(e)). A low magnification view (Fig. 5(f)) of the worn surface corresponding to 3.0 m s^{-1} shows a number of fine debris particles sitting on an otherwise smooth surface. The black layer on the pin surface, visible to the naked eye, was not a distinguishable surface feature in the SEM. Higher magnification view of the same surface showed the grain relief observed earlier. A number of parallel cracks running parallel as well as perpendicular to the sliding direction could also be observed (Fig. 5(g)).

At 4.0 m s^{-1}, during the initial period of high friction sliding, the pin surface was grooved and looked highly deformed. A number of parallel cracks running perpendicular to the sliding direction were also present (Fig. 5(h)). A high magnification micrograph shows a smooth patch surrounded by a number of fragments. This indicates that possibly the worn surface contained a layer formed due to compaction of debris and its disintegration (Fig. 5(i)) caused material removal. At the same speed, when the wear increased with continued sliding (low friction), the worn surface (Fig. 5(j)) consisted of patches which were either smooth or rough. The smooth regions were finely grooved but did not show the degree of deformation seen in Fig. 5(h). The surface had a number of parallel cracks running perpendicular to the sliding direction which, possibly on interaction with each other, caused material removal resulting in the rough patches observed.

Optical micrographs of the disc tracks are presented in Fig. 6(a–d). Original grinding marks could still be seen on the disc surface corresponding to 0.5 m s^{-1} (Fig. 6(a)). At 1.5 m s^{-1} the disc surface was heavily abraded (Fig. 6(b)). While the surface was generally bright and deeply grooved, some grooves were partly covered by grey-coloured deposits. At 3.0 m s^{-1}, as already mentioned, black patches observable by

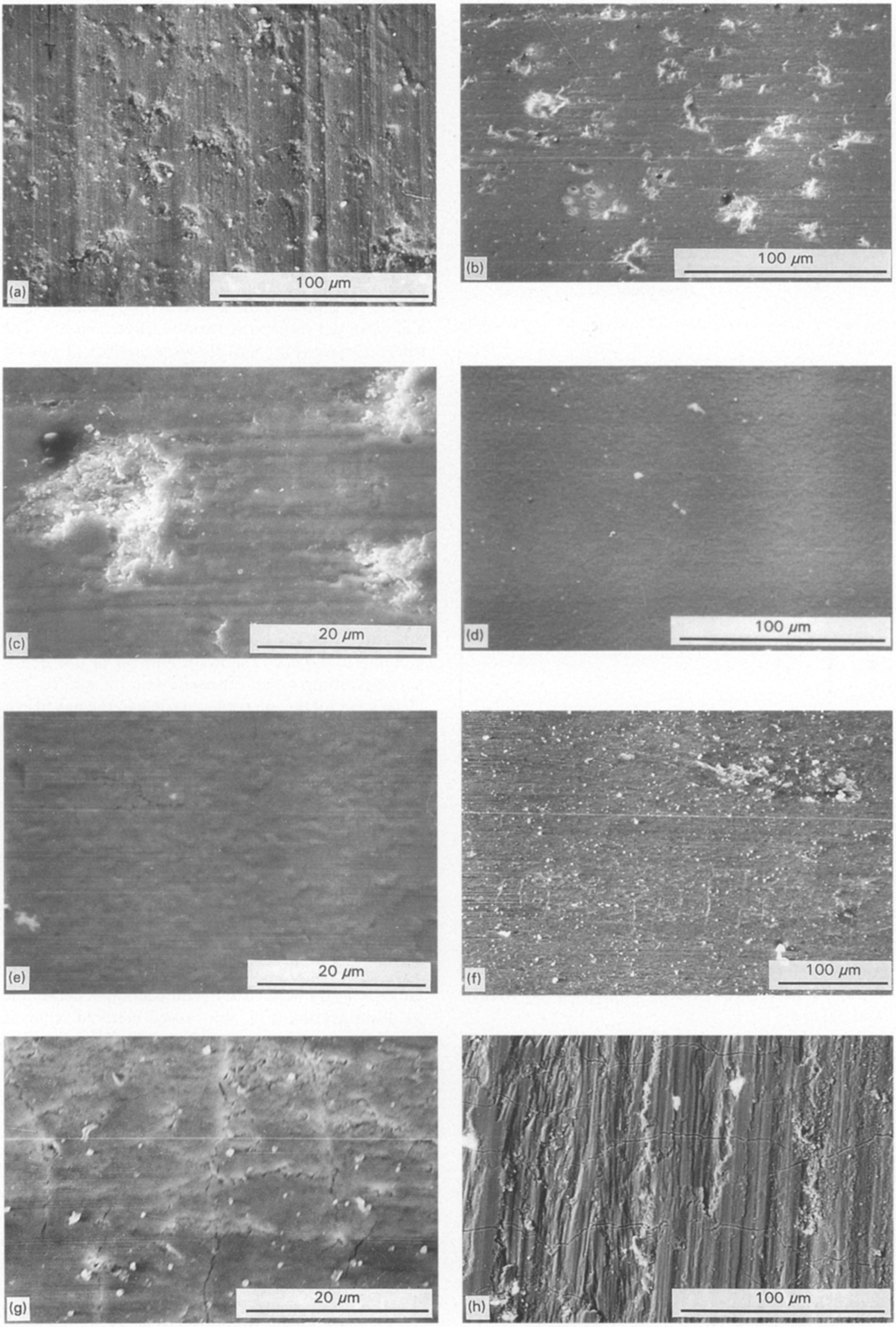

Figure 5 Scanning electron micrographs of pin surfaces. (a) run-in; (b) and (c) slid at 0.5 m s^{-1}; (d) and (e) slid at 1.5 m s^{-1}; (f) and (g) slid at 3.0 m s^{-1}; (h) and (i) slid at 4.0 m s^{-1} during high friction position; (j) slid at 4.0 m s^{-1} during low friction position.

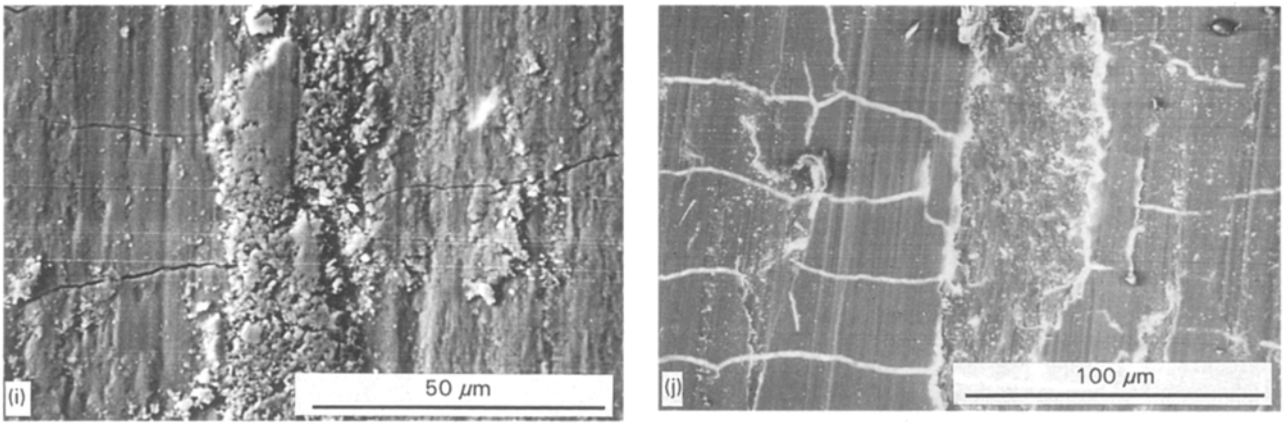

Figure 5 (continued)

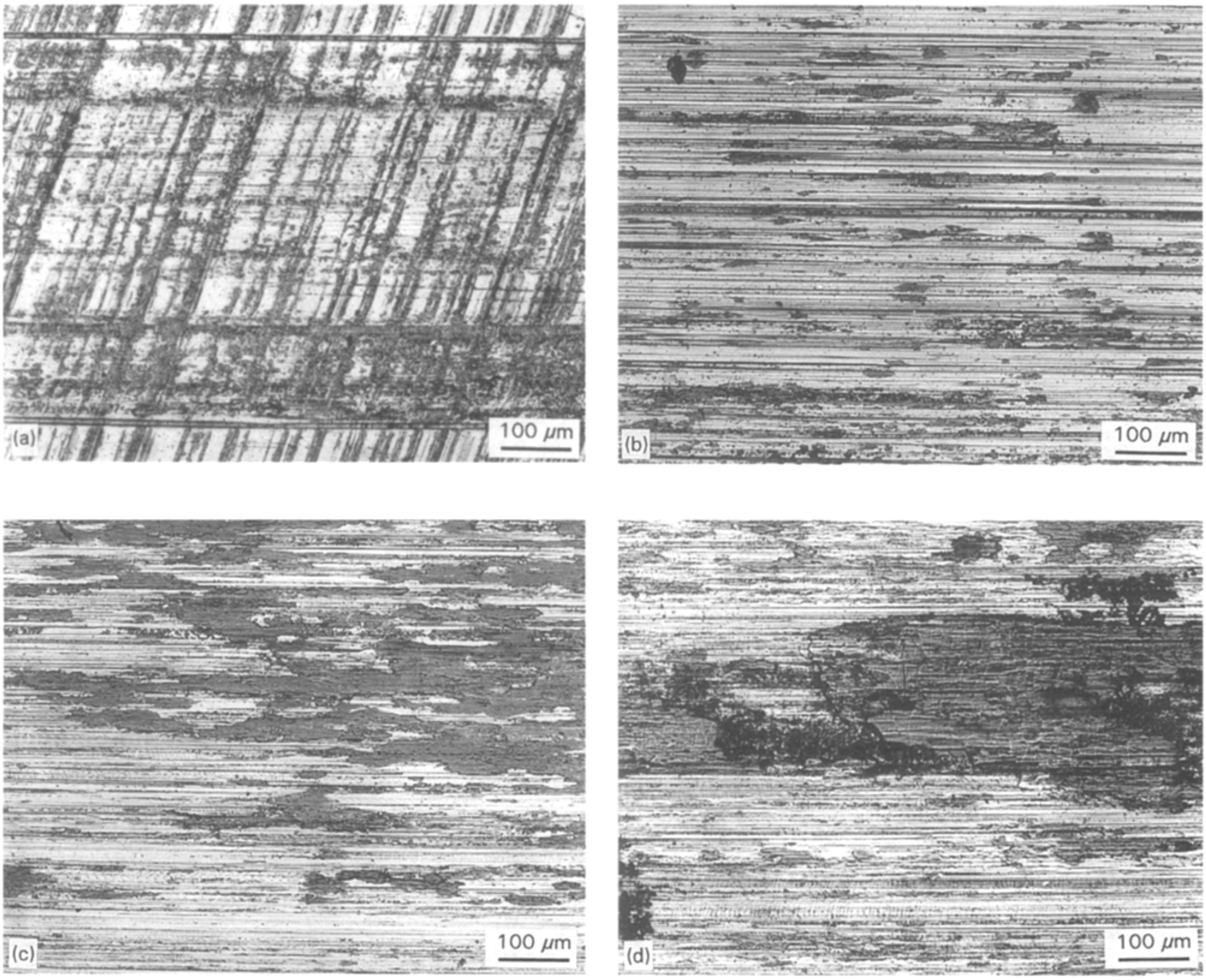

Figure 6 Optical micrographs of disc track at (a) 0.5 m s^{-1}, (b) 1.5 m s^{-1}, (c) 3.0 m s^{-1}, on the black patch and (d) 4.0 m s^{-1} during low friction position.

naked eye appeared on the otherwise bright disc. In the optical microscope, the black patches were seen to be extensively covered by grey deposits (Fig. 6(c)). The region which appeared bright to the naked eye looked similar to that in Fig. 6(b). At 4.0 m s^{-1} during initial periods of sliding, the disc had three types of features. There are thick deposits as shown in Fig. 6(d), patchy areas which looked similar to Fig. 6(c) and abrasion grooves on the rest of the track, which appeared similar to that in Fig. 6(b). During the latter period of sliding (when the wear was very high) all the patchy areas disappeared and only thick deposits on abraded track could be observed.

Debris at low speeds essentially consisted of agglomerates of fine brown particles which are possibly oxides of iron (γ-Fe_2O_3). At 4.0 m s^{-1} two types of debris could be seen; large, flat-shaped debris and small agglomerates of fine particles (Fig. 7(a)). Fig. 7(b)

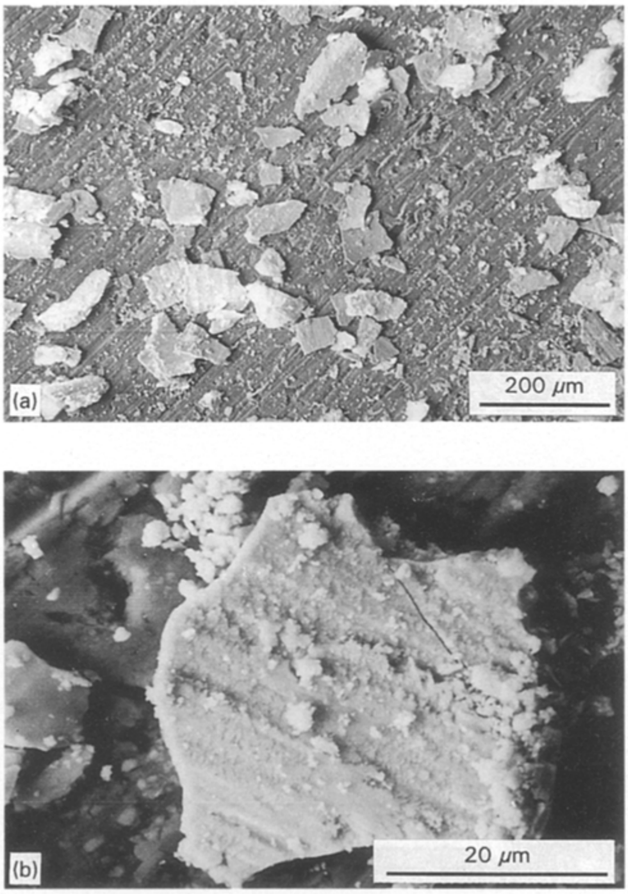

Figure 7 Scanning electron micrographs of debris obtained at 4.0 m s^{-1} at (a) low magnification and (b) high magnification.

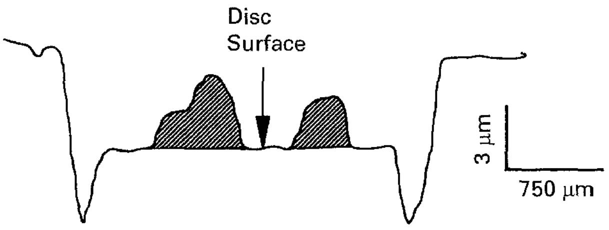

Figure 8 Profile of the disc track obtained at 4.0 m s^{-1} during low friction position. Hatched areas are the ceramic transferred onto the disc.

is a high magnification SEM showing both types of debris.

Profiles of the disc track at 4.0 m s^{-1} were taken to measure the height of the thick deposit, and is shown in Fig. 8; the hatched area in Fig. 8 is the deposit, and its height is about 3.0 μm.

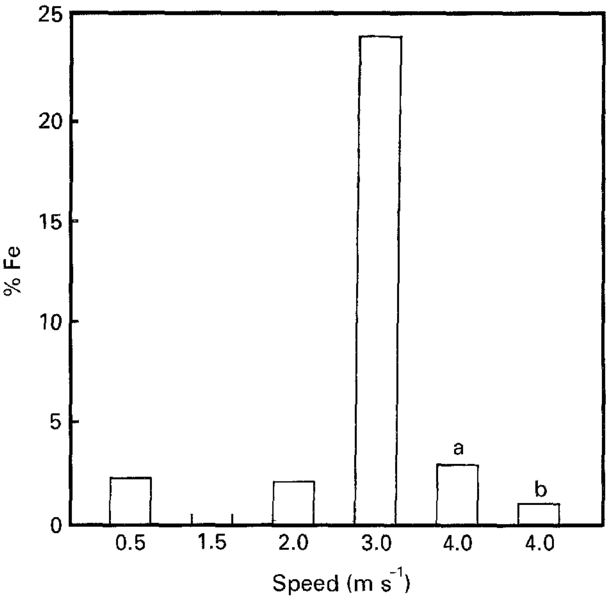

Figure 9 Variation in Fe content on the pin surface with speed. a and b indicate Fe content at 4.0 m s^{-1} during high and low friction position respectively.

3.3. Energy dispersive X-ray (EDXA) analysis

Chemical analysis was carried out by EDXA with ZAF correction. Metal transfer of iron was not observed on the pin surface at any speed. In general, the iron content on the worn surface was very low except at 3.0 m s^{-1}; this is presented in Fig. 9 as a percentage. When analysis was carried out on one of the pits of Fig. 5(b) (at 0.5 m s^{-1}), around 30% iron was obtained whereas outside the pit it was almost zero. This could be due to the entrapment of iron oxide particles in the pit. It implies that the observed iron content at these speeds is probably from such pits. Similar results could be expected from the other surfaces also. The surface of a sample tested at 1.5 m s^{-1} that did not have any pits or rough grooves, gave almost zero content of iron, as expected. At 3.0 m s^{-1} the uniform black layer observed by the naked eye could be a thin layer of oxide of iron, probably that of a high-temperature oxide. The iron content is high for this surface only. However, this oxide layer is so thin and uniformly spread over the entire surface that it could not be identified as a layer by SEM. By carrying out chemical analysis of agglomerates observed on the pin surface obtained at 4.0 m s^{-1} during the initial period of sliding, it could be concluded that they are the compacted debris of zirconia (Fig. 5(i)). Deposits found on the disc at 4.0 m s^{-1} were observed to contain only zirconia.

3.4. X-Ray diffraction studies

X-ray diffraction profiles of an as-received pin, a worn pin surface at 4.0 m s^{-1} high wear position and debris at the same speeds are given in Fig. 10. As-received material contained some monoclinic phase but mostly tetragonal phase, whereas the worn pin surface and the debris contained only tetragonal phase.

4. Discussion

4.1. General

There appears to be a good agreement between the data obtained from tests in which speed was varied sequentially and tests carried out at a constant speed (Fig. 2). Similar observations have been made earlier in the case of Al_2O_3 [18] and Si_3N_4 [19] against steel; this is interesting in view of the observations that for these, wear is dominated by tribochemical interactions, whereas the sliding of YTZP against steel is not, as will be discussed later. The friction characteristics of

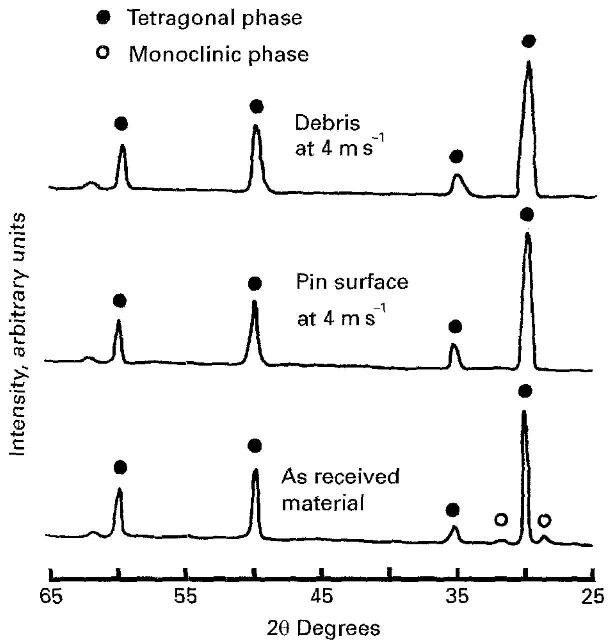

Figure 10 X-ray diffraction pattern (CuK_α radiation) of as received material and debris and pin surface obtained at 4.0 m s^{-1} during low friction position.

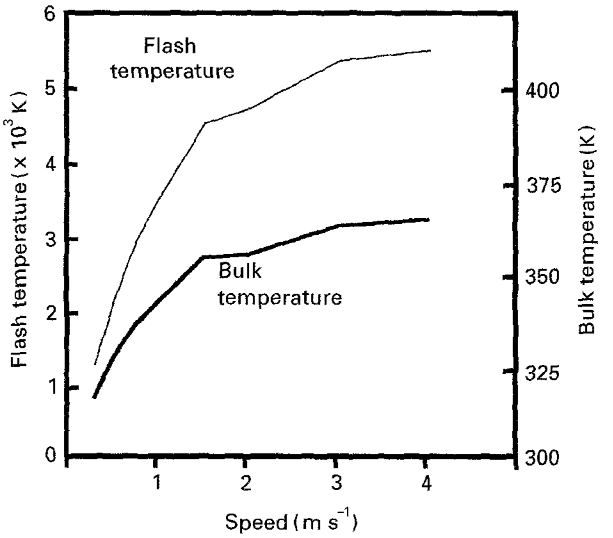

Figure 11 Variation of estimated bulk and flash temperature with speed.

YTZP did not show much sensitivity to variation in speed except at 4.0 m s^{-1} where friction changed with time. The wear characteristics showed that until a certain critical speed was achieved, the measurable wear was zero, and the onset of measurable wear was accompanied by the onset of a red glow at the interface. At the test pressure, the maximum speed that could be employed was 4.0 m s^{-1}.

4.2. The wear mechanism

Although the measurable wear occurred only at 3.0 m s^{-1}, the morphology of the worn surfaces suggested that material was removed to some extent at lower speeds also. The smooth surface morphology observed at 1.5 m s^{-1}, as against that at 0.5 m s^{-1} indicates removal of some layers of material. While pits themselves may be a result of running-in (Fig. 5(a)) the sliding process seems to remove the material by a process of microfracture, making pits disappear. Similar observations have been reported by Aronov and Meysef [20], that there was a reduction in the amount of pits formed with increase in sliding distance at a constant speed, while in the present case it is caused by increasing speed. This latter observation was also supported by observations from direct experiments conducted at various speeds. Thus increasing speed reduces the number of pits and finally, at 1.5 m s^{-1}, surfaces become free from pits (Fig. 5(d)). Grain relief observed at the same speed also indicates preferential pin wear.

Considering the observed change in wear rate between 3.0 and 4.0 m s^{-1}, the wear process can be said to undergo a transition. The important feature of sliding at 3.0 and 4.0 m s^{-1}, is that the surface temperatures are very high. A first order estimation of surface temperatures adopting the model suggested by Lim and Ashby [21] suggests flash temperatures (T_f) of the order of 1200–6000 K (Fig. 11) and bulk surface temperatures of the order of 320–370 K. The values of estimated T_f beyond 1.0 m s^{-1} exceed the melting point of both the mating surfaces and hence the model may not be valid. However, the high T_f (1200 K) at 0.3 m s^{-1} (where the model should be valid) definitely suggests very high interface temperatures at higher speeds. The direct influence of high surface temperature is on plastic deformation, as the hardness and flow stress can be expected to fall [22]. In fact, superplasticity of TZP ceramics has been reported at high temperatues [11, 23]. To account for the characteristic observed wear surface and debris morphologies, the following qualitative physical picture has been suggested.

At 3.0 m s^{-1}, the wear was measurable but small. The surface morphology (Fig. 5(f) and (g)) suggested that most of the debris that could come out would consist of fine particles. Considering that the present conditions are somewhat similar to those employed by Bundschuh and Zum Gahr [8] and further keeping in mind the observed surface rumpling (Fig. 5(g)), and absence of monoclinic phase on both the worn surface and the debris, the mechanism of wear suggested by them can be considered to be operating. According to this model, a thin surface region is heavily deformed, resulting in a t → m phase transformation. Friction-induced thermal heating (depending on the temperature) leads to high temperatures in the surface and allows either the m → t or m → t → c transformation. These phase transformations, accompanied by a volume change produce internal stresses. As a result of the discontinuity in the internal stresses, wear particles may be formed by crack formation and crack propagation.

While the formation of fine particle debris by the above mechanism continues at 4.0 m s^{-1} (Fig. 5(g)), the effect of high temperature-induced plasticity becomes dominant. There is a large degree of surface deformation, surface ploughing (Fig. 5(h)) as well as some material transfer (Fig. 5(i) and Fig. 6(d)). All three together increase the wear rate by orders of magnitude. The high friction probably occurs due to contributions from these various processes. The surface morphology (Fig. 5(j)) suggests an initial large contact area in smooth patches that increases adhesion and material transfer which leaves behind a pit or a crater. Continued sliding at 4.0 m s^{-1} increases the transfer of YTZP to the disc. The increase in wear rate comes about as a result of material transfer, increased deformation due to high temperature and ploughing by hard transferred material and surface cracking.

It is interesting to note that the cracking is similar to that observed by Dufrane [24], which has been attributed to local thermal shock. Rainforth and Stevens [25] have suggested parallel cracking within the grain to be due to phase transformation in bands. In the present case, as the dimensions involved are much bigger than the grain size, it could be due to thermal shock or tensile cracking. The effect of increase in speed is that cracks become more prominent. The average particle size of the debris (Fig. 7(a)) and the intercrack spacing are comparable suggesting that the interaction of these cracks give rise to the type of laminate debris observed.

Considering the extensive deposition of YTZP on the disc surface the reduction in friction (Fig. 1) is possibly caused by the changed interaction i.e. from YTZP pin to steel disc to that between YTZP pin and deposited YTZP. The nature of the imposed deposition process itself is worth considering. From the commencement of sliding at this speed, material transfer from pin to disc increases progressively with time. Under static conditions, in vacuum, a stable diffusion bond has been reported by Cai-Dong Quin and Derby [26] between stainless steel and zirconia. Under sliding conditions diffusion of iron oxide into zirconia (Mg–PSZ) has been reported by Rainforth and Stevens [25], but no material transfer was reported. Under self-mated sliding of Mg–PSZ, however, material transfer was observed by Stachoviak and Stachoviak [27] from the pin on to the disc during initial periods of sliding, when surfaces are smooth, and where diffusional bonding does not exist. In the present case significant transfer is oberved at 4.0 m s^{-1} which also corresponds to very high surface temperatures (Fig. 11). This may be due to enhanced adhesion (between steel and YTZP) at high temperatures. The transfer process accelerates as it changes to that of nearly self-adhesion once the disc track is substantially covered with the ceramic material.

Unlike Al_2O_3 [18], where the steel counterface participated in the wear process, by forming surface layers through chemical interaction, there is no evidence for the role of tribochemical interaction in the case of YTZP. At low speeds wherein metal transfer to ceramic surfaces are generally observed, this is not observed in the case of YTZP. This agrees with the observation of Demizu and Wadabayashi [16], who reported that metal transfer is strongly influenced by surface chemical activity of the metal, and lower surface chemically active metals such as Fe do not show metal transfer. The black surface seen by the naked eye at 3.0 m s^{-1}, and which gave the highest Fe content by EDXA could be from iron oxides, probably a high temperature oxide. The high temperature oxide forms a thin layer, while the low temperature oxide did not, as seen by EDXA analysis of the pin surface at lower speeds, Although at 3.0 m s^{-1} and 4.0 m s^{-1} the calculated interface temperature is very high, even then no observable interaction is noted in the present case.

4.3. Role of transformation

In the present case no evidence of a monoclinic phase was found on the worn surface while the as-received material has 20% monoclinic phase. The absence of the monoclinic phase on the worn-out surface does not rule out any transformation during wear. In metal cutting processes Annamalai *et al.* [12] found no monoclinic phase and they hypothesized that the transformation is reversible i.e., t → m is followed by m → t. Lee *et al.* (28) also do not find any monoclinic phase in the Raman spectra of self-mated worn surfaces of YTZP, but find evidence for the same in debris. Mitra *et al.* (29) have reported that a process such as cutting and polishing takes the transformation to an intermediate level (referred to by them as an x-phase); manifestation of this phase is a noticeable asymmetry in the $(1\,1\,1)_t$ peak. However in the present study, no such asymmetry was found. Considering that the surface temperatures are high at 3.0 m s^{-1} and 4.0 m s^{-1}, it is possible that there is no driving force for a t → m transformation.

5. Conclusions

Dry sliding behaviour of YTZP againstAISI 4340 steel at a pressure of 15.5 MPa and in the speed range of 0.3 to 4.0 m s^{-1} in air led to the following conclusions.

1. No metal transfer on to the pin was observed at any speed.
2. Perceptible wear of the pin starts when the interface becomes visibly red hot.
3. Debris formed in the shape of laminates are due to an interaction area enclosed by tensile cracks observed on the pin surface, whereas fine debris formed was due to intergranular fracture.
4. YTZP pin material was transferred on to the disc at 4.0 m s^{-1} by adhesion and extensive transfer, deformation and ploughing resulted in a transition in the wear behaviour.
5. No m-phase or x-phase could be identified either on the pin surface or in the debris at any speed.

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